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(21) International Application Number: PCT/US91/09611 (22) International Filing Date: 20 December 1991 (20.12.91) (30) Priority data: 632,876 24 December 1990 (24.12.90) US (71) Applicant: GENERAL ELECTRIC COMPANY [US/ US]; 1 River Road, Schenectady, NY 12345 (US). (72) Inventors: PAIK, Kyung, Wook ; 27 Longwood Drive, Clif- ton Park, NY 12065 (US). NEUGEBAUER, Constan- tine, Alois ; 822 Maxwell Drive, Schenectady, NY 12309 (US). (74) Agents: VANDENBURGH, Howard, F.; General Electric Company, International Patent Operation, 1285 Boston Avenue, Bldg. 23CW, Bridgeport, CT 06602 (US) et al.		(81) Designated States: AT (European patent), BE (European patent), CA, CH (European patent), DE (European pa- tent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (Euro- pean patent), IT (European patent), JP, LU (European patent), MC (European patent), NL (European patent), SE (European patent). Published <i>With international search report.</i>
(54) Title: DIRECT BONDING OF COPPER TO ALUMINUM NITRIDE SUBSTRATES (57) Abstract A process for direct bonding a copper film to an yttria-doped aluminum nitride substrate comprises treating the substrate by preoxidation at elevated temperature to create an overlying thin film of Al ₂ O ₃ , followed by step cooling to a lower temperature. A copper foil of thickness between 1.0 and 4.0 microns and generally perforated or otherwise foraminous, is eutectically direct bonded to the substrate by the known direct bond copper (DBC) process. The resultant article exhibits high thermal conductivity, low permittivity and high mechanical strength. The peel strength of the copper film on the AlN substrate exceeds the peel strengths previously attainable in the industry.		

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⁺ Any designation of "SU" has effect in the Russian Federation. It is not yet known whether any such designation has effect in other States of the former Soviet Union.

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DIRECT BONDING OF COPPER TO ALUMINUM NITRIDE
SUBSTRATES

RELATED APPLICATIONS

This application is related to patent
5 applications Serial No. 07/454,547 and Serial No.
07/454,548, both filed December 21, 1989 by
H.F. Webster et al. The former application is
entitled "ENHANCED DIRECT BOND COPPER PROCESS AND
STRUCTURE" and relates to the field of metal bonds,
10 and more particularly, to the field of direct (metal-
metal compound eutectic) bonds. The latter is
entitled "CERAMIC-TO-CONDUCTING-LEAD HERMETIC SEAL"
and relates to the field of electronic packagings and,
more particularly, to hermetically sealed electronic
15 packages in which alumina or beryllia substrates are
bonded with copper through use of a copper oxide
eutectic. Another related application, Serial No.
07/603,495), filed on October 26, 1990, is entitled
"DIRECT THERMOCOMPRESSION BONDING TECHNOLOGY FOR THIN
20 ELECTRONIC POWER CHIP PACKAGES" by C.A. Neugebauer et
al. and is illustrative of techniques for use of
direct bond copper on highly thermally conducting
ceramics. All of the aforementioned applications are
incorporated herein by reference.

25 BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates generally to
direct bonding of copper on ceramic substrates and,
more particularly, to the direct bonding of copper on
30 aluminum nitride.

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Background Information

It has been determined that aluminum nitride (AlN), when sufficiently pure, has a thermal conductivity which is near that of beryllia (BeO). This characteristic makes aluminum nitride a most desirable dielectric for use as a substrate where thermal resistance of the package is a limiting factor. Further, aluminum nitride has a temperature coefficient of expansion (TCE) close to that of a silicon (Si) chip and possesses a thermal conductivity, as previously indicated, approximately ten times that of alumina.

Before a ceramic substrate may be used in the assembling of power devices, it must first be provided with a highly conductive metallization. Normally, copper is chosen for this metallization, in the thickness range of 0.010-0.020 inches. The use of copper metallization in this fashion ensures low electric resistive losses. Direct bonding of metal to ceramic substrates has been known for some time. A significant number of patents have been issued on the subject of direct bond copper (DBC) on ceramic substrates, most notably on alumina or beryllia. See, for example, D.A. Cusano et al. U.S. patent 3,994,430, "DIRECT BONDING OF METALS TO CERAMICS AND METALS", issued November 30, 1976 and G.L. Babcock et al. U.S. patent 3,766,634, "METHOD OF DIRECT BONDING METALS TO NON-METALLIC SUBSTRATES", issued October 23, 1973, both of which are hereby incorporated by reference. DBC on aluminum nitride has been used by the Toshiba Corporation in its line of power modules. More recently, a paper by Kuromitsu et al. of the Mitsubishi Metal Corporation, in the publication of the International Society for Hybrid Microelectronics

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(ISHM)) 1989, entitled "Surface Treatment of AlN Substrate", discusses a metal bond-enhancing surface treatment for AlN substrate. Iwase et al. of the Toshiba Research and Development Center in the publication of the IEEE Components, Hybrids, and Manufacturing Technology, Vol. CHMT-8, No. 2, June 1985, pp 253-258 present a paper entitled "Thick Film and Direct Bond Copper Forming Technologies for Aluminum Nitride Substrate" which relates a certain degree of success in applying DBC technique to a lightly doped (3% yttria) AlN sintered substrate. AlN is one of the most promising candidates as a highly thermally conductive substrate for semiconductor devices. Kuromitsu et al. and Iwase et al. propose a surface treatment for an AlN substrate that would make it suitable to receive most thick film materials that are now used on Al₂O₃ (alumina) substrates. AlN, because of certain superior properties such as high thermal conductivity, low permittivity and high mechanical strength, is indeed a promising candidate as a replacement for alumina, and even beryllia (which is a relatively toxic substance) substrates, if the poor adhesion of films to AlN substrates can be overcome. The instant invention succeeds in overcoming this widely known limitation of metal bonding to an aluminum nitride substrate by providing a method of controlled conversion of AlN to Al₂O₃ to obtain a thin stratum of the oxide on the nitride.

Before discussing the Kuromitsu et al. and Iwase et al. techniques, it is necessary to address recent developments in the preparation of AlN substrates. A cheaper version of AlN of somewhat higher thermal resistivity, but still several times lower than that of alumina, has recently been achieved. The newer AlN is sintered, and is correspondingly doped with a large

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fraction (up to 10%) of yttria which acts as a sintering aid in the fabrication of the substrate. Although resulting in a strong substrate which possesses most of the aforementioned enhanced properties, it has been established that the direct bonding process, most notably DBC, is unsatisfactory under certain conditions; moreover, the earlier published Toshiba recipe, although scrupulously followed, does not result in adherent copper films.

10 An adherent copper (or metal) film, as the term is used herein, means one which has high peel strength when applied on the desired or preferred dielectric substrate. Further, the entire ensemble should possess the high thermal conductivity necessary for
15 dissipation of heat in electronic power modules. Optimally, direct bonded copper (DBC) on beryllia adequately fulfills these criteria; however, unencapsulated beryllia is sometimes considered too toxic for consumer applications. The next favored
20 structure is DBC on AlN, a combination which results in good thermal conductivity but very poor peel strength, as pointed out in the Kuromitsu et al. paper. The least desirable of all the metallized structures is metallization (typically Cu) on Al₂O₃.
25 This latter combination exhibits high peel strength, but possesses poor thermal conductivity with DBC. In fact, the thermal conductivity tradeoff (for the high peel strength) is a loss so great that, in situations requiring good thermal conductivity, use of one of the
30 first mentioned structures is compelling. Since the use of beryllia is preferably avoided in certain applications because of the toxicity question, the DBC as taught herein constitutes a significant improvement over the Toshiba and Mitsubishi AlN substrates
35 relative to the adherence of thin films of copper

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thereto. More specifically, the pre-treatment of the new yttria-doped AlN substrate that is set forth herein results in optimum adhesion when undergoing the DBC process set forth in the aforementioned Babcock et al. patent, for example.

The surface treatment of AlN substrates by Kuromitsu et al. and Iwase et al. comprises, generally, formation of an Al₂O₃ layer on the surface of AlN substrate. Although the Kuromitsu et al. technique also includes mixing alumina with certain glasses, they nevertheless report obtaining sufficient bonding strengths, and, by use of SiO₂, they achieve excellent bond strength. It was observed by Kuromitsu et al. that, when the Al₂O₃ layer is thin, a thicker SiO₂ layer is required to obtain this excellent bond strength. They conclude with the statement that the desirable thickness of Al₂O₃ is thought to be "5 to 8 μ m" in order to retain the excellent thermal conductivity of the AlN substrate. Unfortunately, reliance on the various glasses used in the Kuromitsu et al. process is not always desirable and, even worse, appears to result in a loss in thermal conductivity of the AlN substrate and an increase in outgassing, caused initially by dissolution of the melt glass into the AlN substrate. Thus, by using the published Kuromitsu et al. process, loss of ultimate peel strength is encountered, induced by increased outgassing.

As hereinafter discussed, the instant process for effecting a DBC on aluminum nitride includes a pretreatment of the aluminum nitride that is superior to the Toshiba and Mitsubishi teachings and overcomes the previously mentioned limitations of the technology. The doped AlN has strength superior to intrinsic AlN (substrate) because the yttria enhances

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sintering of the AlN (powder) used to fabricate the substrate. As a consequence, glasses are not required for good adhesion strength and a thinner Al₂O₃ layer (not susceptible to crazing or cracking) permits
5 easier eutectic bonding and provides higher thermal conductivity.

BRIEF SUMMARY OF THE INVENTION

It is generally known that a metal film cannot be bonded to a ceramic substrate unless oxygen is
10 present; that is, without oxygen, generally present as the metal oxide, there can be no eutectic process. Likewise, copper cannot "wet" a ceramic of aluminum nitride unless some form of oxygen is present. In the
15 aforementioned Toshiba process, aluminum nitride is oxidized by lengthy heating in a steam atmosphere. Thus the copper film is bonded to a substrate of Al₂O₃ which is "grown" on the surface of the aluminum
nitride substrate. The Toshiba and Mitsubishi research, however, is unsuitable for the purposes for
20 which the instant invention is required. AlN is softer than most sintered oxide substrates. Poor adherence of Al₂O₃ to intrinsic AlN substrates often compounds the problem of metal (foil) peeling from the
Al₂O₃ that is generally caused by either outgas
25 blistering of the foil or delamination from a crazed (largely cracked) Al₂O₃ layer. Use of glasses (SiO₂, PbO) strengthens the Al₂O₃ layer, but does little for the unreinforced AlN substrate. Further, outgassing
can actually be increased, as pointed out by Kuromitsu
30 et al. In arriving at the present invention, it was realized that a stronger AlN substrate could obviate need for a thick Al₂O₃ layer and for use of glassy additives with their concomitant increase in
outgassing. A thinner layer is also less susceptible

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to crazing or cracking and is better for good thermal conductivity also.

Quite unexpectedly, it was found that it is possible to achieve optimum processing conditions in which the peel strength of the DBC foil on the aluminum nitride substrate exceeds 14 pounds per inch (measured across the width of the sample), thus overcoming the low-peel strength problem previously associated with doped AlN. These optimum conditions, for the yttria-doped AlN, involve oxidizing the nitride surface for 30 minutes at 1220°C in an atmosphere of N₂/O₂, 10:1 by volume, in the presence of water vapor. In the subsequently performed DBC operation, a perforated foil is used to prevent blistering in the film caused by any latent or spurious outgassing from the preoxidized AlN substrate. In lieu of the perforated copper film, the various earlier techniques for avoiding delamination due to outgassing or other thermal stress can also be used, such as the "lacy" construction of thermal compression bonded films described in application Serial No. 07/603,495 or the grooving techniques disclosed in U.S. Patent No. 4,409,2278, issued on October 11, 1983 to Eric P. Jochym and entitled "BLISTER-FREE DIRECT BONDING OF METALS TO CERAMICS AND METALS".

DETAILED DESCRIPTION OF THE INVENTION

Intrinsic AlN is doped with up to 10% yttria in order to effect a strong substrate by sintering. A pretreatment is then conducted on the AlN substrates by first establishing the proper gaseous mixture for an environment in which to conduct the oxidation of the aluminum nitride. A mixture of 0.3 SCFH (cubic feet per hour at standard conditions) of oxygen gas

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and 3.0 SCFH of nitrogen gas ($O_2:N_2=1:10$) flowed together, is supplied to a water baffle which is adjacent a conventional tube furnace and which is held at room temperature. In establishing these conditions, it has been determined empirically that a higher oxygen amount would produce too rapid an oxidation reaction, and vice versa. A water-bubbled mixture of the aforementioned gas constituents is then introduced to the furnace which has an open end at the side opposite the point of introduction. Initial flow of the gas mixture to the water baffle controls the water vapor in the furnace chamber thereafter.

The temperature of the furnace is initially set to a temperature regime of 1200°C to 1250°C , in order to establish the temperature initial conditions (T_{ic}). After the temperature stabilizes, samples (i.e., various substrates) are loaded into a quartz carrier (i.e., boat) and inserted into the quartz tube that passes through the furnace. Oxidation time is measured from the time the furnace temperature is reestablished, following specimen loading at T_{ic} .

It has been further determined that optimum time-temperature process conditions for oxidation are 30 minutes at 1220°C . Oxidation time may vary, depending on temperature. Overoxidation at a higher temperature or longer time creates a thicker alumina surface on the AlN substrate, resulting in surface cracks which will inevitably prevent good adhesion of copper films on the AlN. Subsequently, upon removal from the furnace, the specimens are allowed to step cool in order to avoid chill-induced cracking or crazing in the newly created alumina surface, due to a mismatch in temperature coefficient of expansion between AlN and Al_2O_3 . This step cooling may be performed by moving the sample from a central location

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in the furnace to a location in the furnace that is near the edge of the furnace (so as to reside at an ambient temperature of over 700°C) for a period of about 3-5 minutes, until the sample has changed in color from orange to gray. At this time the sample can be relocated outside of the furnace and allowed to cool for a period of about 3 minutes. The sample is then removed from the quartz tube. To achieve maximum adhesion, all conditions are carefully controlled to create an alumina surface thickness on the AlN ranging from 1-4 microns, with an optimum (for obtaining very high peel strength) being between 2 and 3 microns, and most preferably 2 microns.

After the alumina-layered AlN substrates are prepared and cooled, DBC processing is conducted on them using a perforated copper foil in order to prevent foil blistering which might be caused by outgassing from the AlN substrate. Alternatively, lacy construction, as taught in application Serial No. 07/603,495 or surface modification (grooving) as taught in U.S. Patent No. 4,409,278 may be effected, resulting in the same favorable adhesion characteristics exhibited by the perforated copper foil. Analysis has shown the peel adhesion strength of 5 mil thickness copper foil on AlN to be 14.5 pounds per inch, which is almost 80% of the peel strength of direct bonded copper to alumina.

The table below indicates the adhesion strength of copper films on standard 2" x 2" 50 mil thick yttria doped AlN substrates purchased from Carborundum Company, Electronic Materials Group, Substrates Division, 2050 Cory Drive, Sanborn, NY 14132, at the various AlN surface treatment conditions. At the maximum adhesion condition, the observed thickness of the surface alumina is 2.0 microns, significantly

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thinner than either of the 5.0-8.0 microns reported in the aforementioned Kuromitsu et al. paper, or the 4.0 microns reported in the aforementioned Iwase et al. paper and attained only as a result of direct bonding Au to AlN through use of other metallics. Thus high peel strength is acquired without use of additional metallics and is consistent with the uncrazed, sparser and thinner Al₂O₃ lamina achieved by the invention. This new laminate demonstrates that the thermal conduction of AlN dominates the Al₂O₃, achieving a metallized ceramic substrate not quite possessing all the attributes of DBC-on-beryllia, but of significantly better thermal character than either of the prior known DBC-on-AlN or DBC-on-alumina. Also significant is the fact that the present invention realizes a combination of highly conductive metallization-on-ceramic which exhibits high thermal conductivity, low permittivity and, because of the increased yttria doping of the AlN, high mechanical strength.

The following table details more extensively the empirical results of the invention:

Table Of Peel Strength Of Copper Film
(5 mil Thickness) That Has Been
Applied By DBC On Preoxidized Yttria-Doped AlN
Substrates At Specific Conditions

	<u>Conditions</u> <u>(lbs./in.)</u>	<u>Peel Strength</u>
	<u>As-received</u>	1.2
30	<u>1 O₂ + 100 N₂ (1:100)</u>	
	1220°C/1 hr.	3.7
	1220°C/16 hrs.	3.9
	1250°C/1 hr.	9.2

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10 O₂ + 100 N₂ (1:10)

	1170°C/ 1 hr.	10.0
	1190°C/25 mins.	10.4
	1190°C/45 mins.	10.1
5	1220°C/10 mins.	2.9
	1220°C/20 mins. *	11.7
	1220°C/25 mins. *	12.0
	1220°C/30 mins. *	14.5
	1220°C/35 mins.	9.1
10	1220°C/40 mins.	7.2
	1220°C/ 1 hr.	7.4
	1250°C/10 mins. *	11.3
	1250°C/15 mins.	8.6
	1250°C/20 mins.	7.7
15	1250°C/ 1 hr.	5.7

20 O₂ + 100 N₂ (1:5)

	1220°C/10 mins.	7.7
	1220°C/20 mins.	9.8
	1220°C/30 mins.	8.6
20	1220°C/ 1 hr.	6.1
	1250°C/10 mins. *	11.6
	1250°C/20 mins. *	10.0

Pure O₂

	1250°C/10 mins.	9.6
25	1250°C/20 mins.	6.1
	1250°C/30 mins. *	10.3
	1250°C/40 mins.	4.6

Air (O₂:N₂ = 1:4)

	1250°C/30 mins. *	11.8
30	1250°C/ 1 hr.	5.5
	1250°C/ 1.5 hrs.	12.8

For comparison purposes:

Alumina substrate 18.4

* regime of consistently optimal results

35 It may be seen from the above data that optimum results of 14.5 lb/in. peel strength are achieved at 1220°C with a treatment time of 30 mins., in an atmosphere of water vapor and O₂:N₂ in a ratio of

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1:10. A desired surface texture of the deposited alumina that could best be described as "spongy", i.e., elastic and with a porous property that provides a good bonding interface, is achieved, thus effecting a surface modification analogous to the "grooving" of Jochym ('278). The spongy characteristic derives from a hydration of the alumina which varies in dependence upon the amount of water present in the reaction gas mixture. Thus the strength of the bond depends on the Al_2O_3 properties, thickness and texture, in a particular type of surface layer which is attained on an AlN substrate by the instant process. The required thickness of the Al_2O_3 layer is achieved only under the carefully controlled conditions discussed herein, and as noted in the above table.

In an alternative embodiment for producing the AlN substrates coated with 2.0 microns thickness of alumina to undergo DBC, yttria-doped AlN substrates are loaded into a Pyrogenic steam furnace at a temperature of 700°C in a nitrogen and oxygen atmosphere maintained at a flow rate of 1 liter(l)/min. O_2 and 8 l/min . N_2 . Furnace temperature is then ramped up to about 1150°C in 1 hour. The furnace atmosphere is then stabilized to pure O_2 , and a steam environment is next created by introducing O_2 at a 2 l/min . flow rate and H_2 at a 3.5 l/min . flow rate. The samples are held in the steam environment for about 1 hour. The H_2 flow is then shut off and the O_2 flow is increased to a 4 l/min . rate for 5 minutes to drive off the H_2 . The samples are next step cooled to about 700°C in 2 hours in a nitrogen atmosphere maintained by a nitrogen flow rate of 8 l/min ., and then allowed to cool to room temperature. An AlN substrate fabricated in this

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manner has been found to exhibit a 14 lb./in. peel strength for copper film of 5 mils thickness.

While only certain preferred features of the invention have been illustrated and described herein,
5 many modifications and changes will occur to those skilled in the art. It is, therefore, to be understood that the appended claims are intended to cover all such modifications and changes as fall within the true spirit of the invention.

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What is claimed is:

1. A method for direct bonding a high peel-resistant metal conductive film to a yttria-doped aluminum nitride substrate comprising preoxidizing the substrate in order to create thereon a thin layer of
5 Al_2O_3 of thickness between 1.0 and 4.0 microns, and thereafter direct bonding a foraminous, thin copper film to said thin layer of Al_2O_3 using a direct bonding copper eutectic process.

2. The method of claim 1 further comprising preoxidizing the substrate by heating it to a temperature of between 1200°C and 1250°C for between 10 and 30 min. in an atmosphere of water vapor and
5 $\text{N}_2:\text{O}_2$ in a proportion in the range of about 10:1 to about 4:1, and thereafter step cooling said substrate.

3. The method of claim 2 wherein said step cooling is performed in atmosphere.

4. The method of claim 2 wherein the layer of Al_2O_3 is between 2 and 3 microns in thickness and the eutectic is comprised of copper-copper oxide.

5. The method of claim 4 wherein said step cooling is performed in atmosphere.

6. The method of claim 2 wherein the layer of Al_2O_3 is 2.0 microns thick and the eutectic is comprised of copper-copper oxide.

7. The method of claim 6 wherein said step cooling is performed in atmosphere.

8. A method for obtaining peel resistant direct metal bonds on an aluminum nitride substrate comprising pretreating the substrate by slowly

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oxidizing it in an atmosphere of water vapor and $N_2:O_2$ mixture in the range of about 10:1 to about 4:1 for between 10 and 30 minutes at a temperature in the range of 1200°C to 1250°C, step cooling the oxidized substrate, and eutectically direct bonding thereon a thin, foraminous copper foil.

9. The method of claim 8 wherein the pretreating step comprises oxidizing the substrate slowly until an Al_2O_3 layer between 2 and 3 microns in thickness is formed thereon with a thin, irregular surface texture conducive to DBC adherence.

10. The method of claim 8 wherein the pretreating step comprises oxidizing the substrate slowly until an Al_2O_3 layer 2 microns in thickness is formed thereon with a thin, irregular surface texture conducive to DBC adherence.

11. A method for making a DBC-on-AlN article that possesses high peel strength with high thermal conductivity, low permittivity, and mechanical strength greater than intrinsic AlN sintered articles, comprising the steps of:

oxidizing a yttria-doped AlN substrate in a Pyrogenic steam furnace at a temperature of about 1150°C for about 1 hour to obtain an Al_2O_3 layer on surfaces of the substrate of about 2 microns in thickness; and

direct bonding eutectically a foraminous copper foil on at least one Al_2O_3 -covered surface of the substrate.

12. The method of claim 11 wherein said oxidizing is performed in an atmosphere of hydrogen and oxygen introduced at flow rates of 3.5 l/min. and 2 l/min.,

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respectively, and is followed by step cooling to about
5 700°C in about 2 hours in nitrogen prior to said
direct bonding.

13. An article of manufacture comprising a
yttria-doped AlN substrate bearing thereon an Al₂O₃
layer and a copper layer further bonded eutectically
on said Al₂O₃ layer so that said Al₂O₃ layer is
5 between the copper layer and the doped AlN substrate,
said AlN substrate being doped with up to 10% yttria
and said Al₂O₃ layer having a thickness in the range
of 1-4 microns.

14. The article of claim 13 wherein said Al₂O₃
layer thickness is in the range of 2 to 3 microns.

15. The article of claim 14 wherein said copper
layer is foraminous.

16. The article of claim 13 wherein said Al₂O₃
layer thickness is 2.0 microns.

17. The article of claim 16 wherein said copper
layer is foraminous.

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INTERNATIONAL SEARCH REPORT

International Application No. PCT/US91/09611

I. CLASSIFICATION AND SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
IPC (5) : B23K 1/20; B32B 15/04, 18/00, 15/20		
US. CL. : 228/122, 123, 198, 263.18; 428/336, 469, 472, 697, 698, 701, 702		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System :	Classification Symbols	
U.S.	228/122, 123, 198, 263.18; 428/336, 469, 472, 697, 698, 701, 702	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X Y	IWASE ET. AL., "Thick Film and Direct Bond Copper Forming Technologies for Aluminium Nitride Substrate", June 1985, pages 253-258, Vol. CHMT-8, No. 2. See page 255, line 1, lines 18-29, page 256, lines 11-24.	1, 13 2-12, 14-17
Y	US, A, 4,659,611 (IWASE ET. AL.) 21 APRIL 1987; See Column 7, lines 14-27, Column 5, lines 12-24; Column 6, lines 25-38.	1-12
Y	US, A, 4,892,703 (IIO ET. AL.) 09 JANUARY 1990, See Table 2.	1-12
<p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search		Date of Mailing of this International Search Report
26 FEBRUARY 1992		19 MAR 1992
International Searching Authority		Signature of Authorized Officer
ISA/US		Archene A. Turner